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Automatic Recording in Serial Colorimetric Analysis

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SERIAL colorimetric analysis, as encountered in chromatography, countercurrent analysis, zone electrophoresis, etc., should be greatly facilitated through the use of an automatic recording device. To be most useful, such a device should provide point recording at fixed wave length, uniform spacing of points along the Y-axis of the recorder, and push-button control for intermittent operation. Commercially available automatic recording spectrophotometers are not best suited to this application, because, in general, they are more specifically designed for continuous recording at variable wave length.

To devise an instrument having the requisite features, a Brown Electronik strip chart recorder was modified for use with a Model 401-E Lumetron photoelectric colorimeter, by applying the voltage output of the photocell of the colorimeter to the input terminals of the recorder and modifying the chart drive mechanism of the recorder to provide for intermittent operation and uniform spacing of points. To produce a linear response with respect to the absorbance of samples being measured, the linear slide-wire of the recorder was replaced with a logarithmic slide-wire. Appropriate resistance was also introduced into the circuit to render the characteristics of the recorder the same as those of the microammeter with which the colorimeter was originally equipped. A double-throw switch permits use of either the recorder or of the original microammeter.

The Model 401-E Lumetron photoelectric colorimeter was selected for incorporation in the automatic recorder, because of the convenience of its use of test tubes as absorption cells and of a sliding tube carrier which accommodates both reference and sample tubes. With modifications in circuit, other photoelectric colorimeters or spectrophotometers could also be used.

The automatic recorder thus devised provides dot recording of absorbance as X-points spaced at equal intervals along the Y-axis. The X-axis in this instance is in the direction of pen travel and represents the dependent variable; the Y-axis, in the direction of chart travel, the independent variable. The scale of the X-axis is graduated 0 to 20 along the center 10 inches of the chart, corresponding to absorbance values from 0 to 2. Intervals of chart movement along the Y-axis are adjustable between 0.1 and 0.2 inch. Successive points along the Y-axis will generally refer to consecutive sample numbers. Minneapolis-Honeywell chart No. 5839-N, which is used with the recorder, is graduated in tenths of an inch, and permits readings of absorbance to 0.01 unit and estimates to 0.001 unit.

A photograph of the complete assembly is shown in Figure 1. The pattern appearing on the chart in the recorder was obtained in a simulated test and illustrates the practical application of the instrument.

An assembly bearing a marked resemblance to the present was described very recently by Solomon and Caton [Solomon, A. K., Caton, D. C., ANAL. CHEM. 30, 295 (1958)].

DESCRIPTION

Recorder. The recorder used is a modified Brown Electronik strip chart potentiometer, Model 153 × 65. This is a two point synchro-print instrument with a pen speed of $4^1/_2$ seconds for full scale pen travel and −1- to +21-mv. reversed range.

Chart Drive Mechanism. The modification of the chart drive mechanism is shown in Figure 2.

The standard change gears are removed, disconnecting the original synchronized drive linkage between the drive motor and the chart roll. A new slip clutch and gear assembly, A, is installed and meshed with the standardizing gear, B. This clutch has a stop pin, C, which engages with a

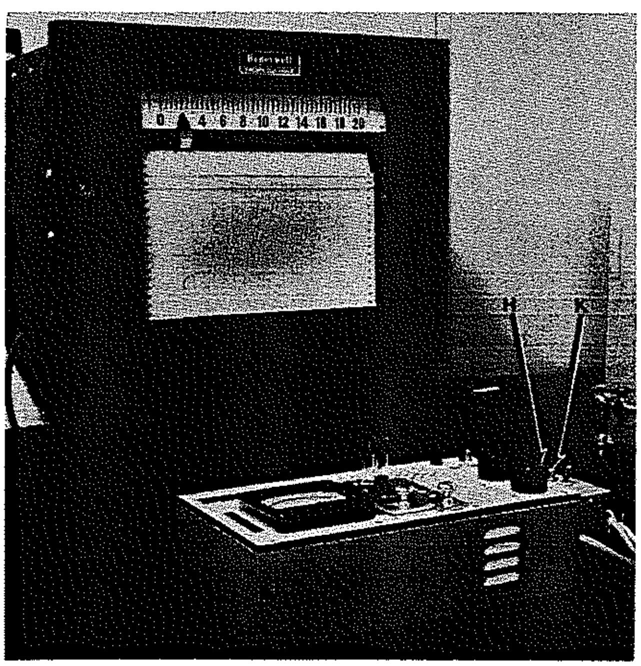

Figure 1. Automatic recorder assembly with pattern of simulated test

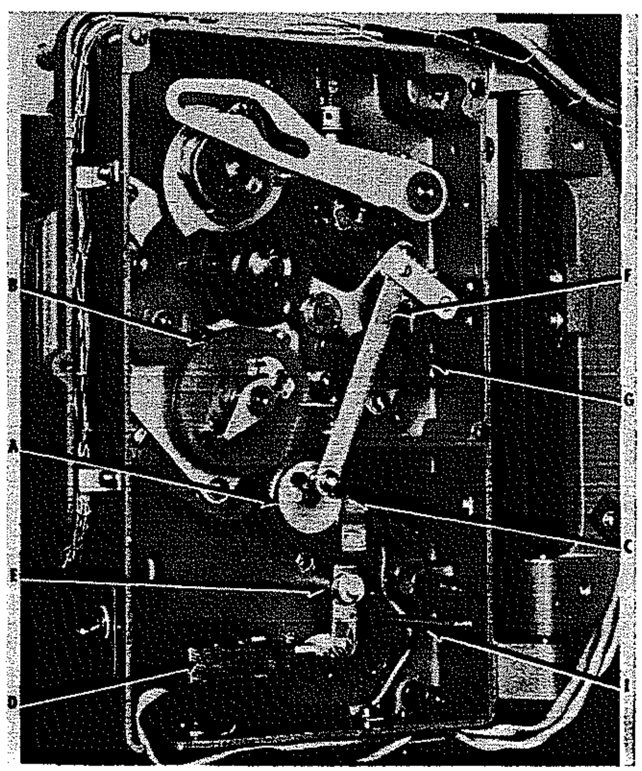

Figure 2. Modification of chart drive mechanism

solenoid and pawl mechanism, *D*, *E*, allowing the clutch to slip at all times except when chart advance is desired. A connecting rod, *F*, transfers the adjustable crank motion on the slip clutch to a roller ratchet, *G*, on the chart drive shaft.

To advance the chart, the chart advance button (*H*, Figure 1) is depressed, then released. Depressing the button energizes the solenoid, which moves the pawl away from the stop pin; releasing the button de-energizes the solenoid and allows the spring return (*I*, Figure 2) to pull the pawl back to its original position. Unless the button is held depressed, the slip clutch makes only one revolution, coming again to rest on the pawl. During the movement of the slip clutch, the connecting rod goes through one cycle and advances the chart. The complete cycle takes about 2 seconds.

If at any time it is desired to return the chart drive to its original synchronized drive, it is necessary only to remove connecting rod *F* and replace the proper change gears.

Print Mechanism. The Brown recorder is factory-equipped with a print holdout mechanism which prohibits printing during standardization of the measuring circuit.

In the present instance, the automatic standardizing is removed from the recorder to prevent its functioning during a print cycle. Advantage is taken of the print holdout mechanism in an arrangement which allows push-button printing, by springloading the hold-out pin and using a solenoid (*J*, Figure 3) for releasing the tension. When the button (*K*, Figure 1) is depressed, the solenoid is energized, the tension on the print hold-out pin is released, and the plunger closes a microswitch (*L*, Figure 3) which keeps the solenoid energized until the recorder comes to balance and the neutral detector starts the print cycle. After the print wheel has come in contact with the chart and started its return stroke, a second microswitch (mounted on reverse side of chassis plate shown in Figure 3) is contacted, releasing the solenoid and again applying tension to the holdout pin. The print mechanism retains its "ready" position until the print button is again depressed.

If the print button is held down, the instrument will function as a standard synchro-print recorder. To convert the mechanism back to a standard synchro-print recorder, it is necessary only to remove the spring tension on the hold-out pin.

Range Function Modification. The modification of the range function consists of the installation of a dual 40-db. logarithmic potentiometer (*M*, Figure 3), one to replace the linear slide-wire with which the recorder is originally equipped, the other to function as a gain control for the servoamplifier. The latter is required, because of the wide range in which the instrument must function. At the low end of the range high gain is needed, because the recorder is, in effect, working at a very small full scale range, and at the high end the full range is very large and the recorder tends to hunt.

This specially designed dual potentiometer is installed in such a way that the high end of the function is at the left end of the recorder scale, where 16.57 mv. produces zero deflection, and the low end of the function is at the right end of the recorder scale, where approximately 0.166 mv. produces full-scale deflection.

Circuits. The electrical circuit for the push-button chart drive and print mechanisms is shown in Figure 4; that for the modification of the Lumetron colorimeter for use with the recorder is shown in Figure 5. The 240- and 1000-ohm resistances shown in the latter figure are present in the original colorimeter circuit and serve to vary the sensitivity of the colorimeter microammeter. These resistances perform the same function for the recorder although introduction of a 135-ohm resistor at the location shown is necessary for matching the

Table I. Response of Recorder to Input Voltage

Absorbance Scale Reading	Required Input Voltage, E Mv.	Log $E_0{}^a/E$	Recorder Error, %
0.000	16.57[a]	0.0000	0
0.100	13.11	0.1018	−2
0.200	10.34	0.2048	−2
0.400	6.56	0.4024	−1
0.600	4.22	0.5940	+1
0.800	2.61	0.8027	0
1.000	1.68	0.9940	+1
1.200	1.075	1.184	+1
1.400	0.687	1.382	+1
1.600	0.424	1.582	+1
1.800	0.278	1.775	+1
1.900	0.229	1.859	+2

[a] E_0 = 16.57 mv.

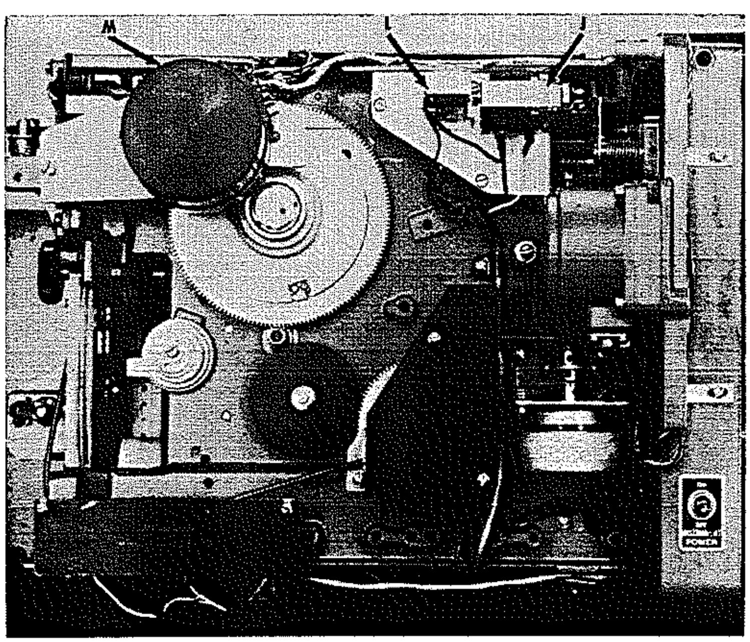

Figure 3. Modifications of print mechanism and of range function

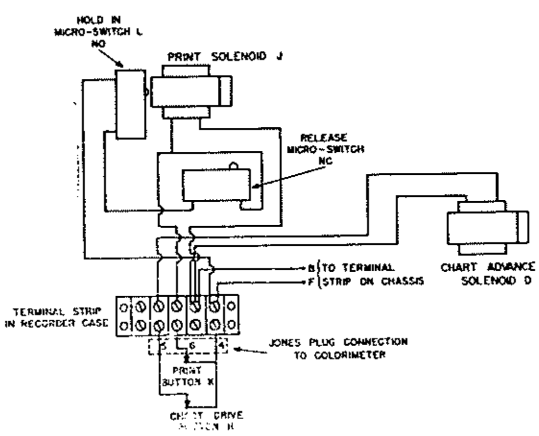

Figure 4. Electrical circuit for chart drive and print mechanisms

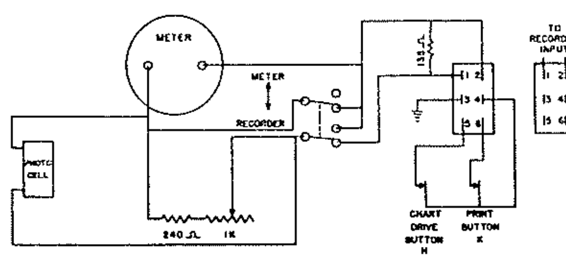

Figure 5. Electrical circuit for modification of Lumetron colorimeter for use with Brown strip chart recorder

characteristics of the recorder with those of the microammeter.

Performance. The input voltages required to produce different absorbance scale readings are shown in Table I. The voltages were supplied by a Type 8662 Leeds & Northrup potentiometer. For perfect response, the absorbance scale reading should equal the logarithm of the ratio of the voltage producing zero scale deflection, E_0, to that producing the observed reading, E. The errors in the observed readings at different positions on the scale, shown in the last column of the table, are not greater than 2%.

The reproducibility of the distance of pen travel was found to be ±0.003 absorbance unit at an absorbance of 0.100, ±0.003 unit at an absorbance of 0.900, and ±0.013 unit at an absorbance of 1.700.

The above levels of accuracy and of reproducibility will be adequate for practically all work for which the use of the present type of instrument is indicated.

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